# THE ELECTROSTATIC PRECIPITATION OF SOAPS FROM OILS

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## A THESIS

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PRESENTED BY

**G. M. DOWSE AND M. O. BRUECKNER**

TO THE

PRESIDENT AND FACULTY

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IN

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# THE ELECTROSTATIC PRECIPITATION OF SOAPS FROM OILS.

This treatise deals primarily with the work done to devise some means of precipitating a solid in a liquid by electrostatic methods. The application of such a scheme to the industries would be applicable in precipitating the soap formed during the refining of nearly all the edible oils, thereby eliminating a filtration operation which is now necessary to remove the soap stock formed upon the addition of the caustic Soda in the preliminary refining operation. Filer-pressing the refined oil to remove the soap stock is a comparatively slow process and the work which was carried out in the preparation of this paper was

done to try and find some scheme featuring continuous, economical and rapid removal of the solid matter formed in the oil during the refining period.

A secondary consideration was made with reference to the refining of the particular oil used. Raw Cotton-Seed oil was used for all the experiments, the refining being conducted in connection with the other work. An attempt was made to determine what effects temperature, weight of caustic Soda used, and the strength of the caustic Soda solution had upon the nature of the precipitated soap and the time required for complete precipitation of the soap stock formed. No attempt was made to determine the refining losses or to determine the effect of the above variations upon the refining losses.

An analysis was made of the raw oil to determine the percentage of free fatty acid present. The method of analysis used was as found in Sherman's Organic Analysis, Chapter V111, Page 147. From the information thus obtained, the approximate weight of caustic Soda was calculated which would neutralize the free fatty acids in the oil, and after knowing this, a series of runs were made with varied conditions as to weight of caustic Soda used, above or below the calculated amount, time of run, temperature of the oil at different periods and strength of the caustic Soda solution when added to the oil. The first trials were made with a 10 degree Baume' solution of caustic Soda, the amount used being varied and also the temperatures at different parts

# OIL REFINING APPARATUS

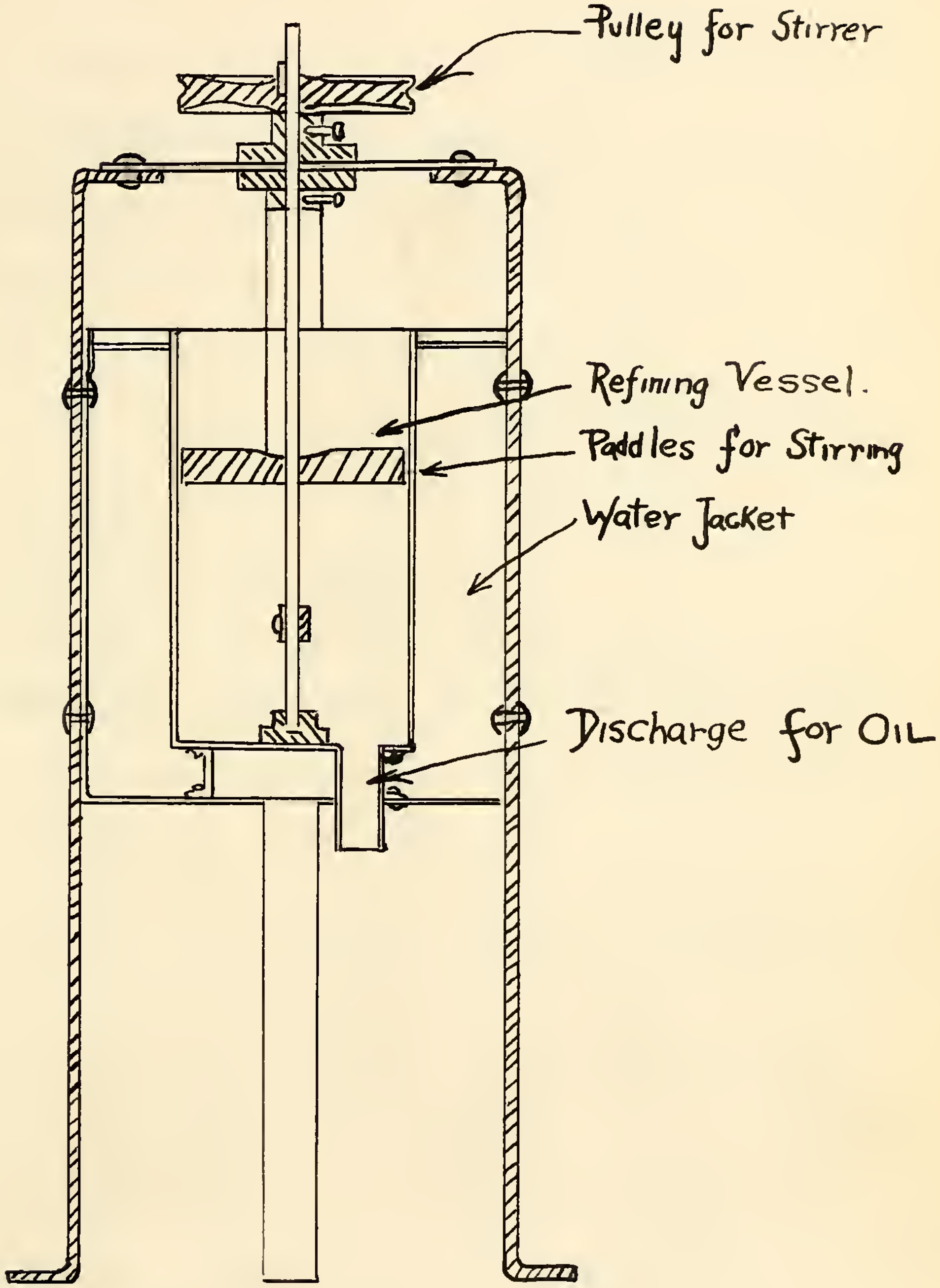

Fig I.

SCALE – 3" = 1 FT.

of the process being varied. Solutions of 14,16 and 20 degrees Baume' were used in the same manner under similiar conditions as for the first trials. The nature of the precipitated soap stock was noted in each case and also the rapidity with which it settled and whether or not it was firm and coherent.

The amounts of oil and Caustic Soda used, the conditions under which the trial was made and the observations made are recorded later. The deductions made from the results obtained by applying the variations noted in a preceding paragraph are also given. The form of apparatus used for refining the oil is shown in Figure 1.

In the titration for free fatty acids, o.o1309 grams of NaOH were used for 4.6571 grams of oil, or 1 gram of oil requires

o.0028 grams NaOH to neutralize the free acid. The Specific Gravity of the oil was 0.92 and 500 cc were used in each trial. The theoretical amount of NaOH required for 500 cc of oil would be 1.288 grams, but this amount was varied as shown in the following data.

## VARIATIONS OF CONDITIONS IN REFINING THE RAW COTTON-SEED OIL.

SAMPLE #1.

Weight of oil used -------460 grams.

Strength of caustic ------ 10 degrees Baume'.

Volume of caustic used --- 30 cc.

Conditions under which run was made;

The NaOH was added when the temperature of the oil was 25 degrees C. After the NaOH was added, the oil and caustic were

agitated for twenty minutes. The temperature was gradually raised so that in about fifteen minutes the temperature was 45 degrees C. The oil was kept at this temperature for two hours and then cooled to room temperature.

The soap stock formed was firm and coherent and settled rather rapidly and left a clear oil. The color of the oil was fairly good.

SAMPLE #2.

Weight of oil used -------460 grams.
Strength of **caustic** used-- 10 degrees Baume'.
Volume of caustic used --- 20 cc.

The conditions under which the run was made were the same as for sample #1.

The soap stock formed was not as firm as in the first trial and did not seem to settle rapidly. The color of the oil

was not as good as in the previous run.

SAMPLE #3.

Weight of oil used -------460 grams.

Strength of caustic used - 10 degrees Baume'.

Volume of caustic used --- 30 cc.

Conditions under which the run was made;

The caustic solution was added at 25 degrees C. This temperature was maintained throughout the entire run.

The soap stock formed seemed to settle rapidly and the color of the oil seemed to be better than in previous trials.

SAMPLE #4.

Weight of oil used -------460 grams.

Strength of caustic used --10 degrees Baume'.

Volume of caustic used --- 30 cc.

Conditions under which the run was made;

The caustic solution was added when the temperature of the oil was 25 degrees C. The oil was agitated for twenty minutes at this temperature and then heated slowly to 70 degrees and held there for two hours.

The refined oil seemed to be cloudy and from appearances, it seemed that some of the oil had been saponified by the caustic.

From the above data,it seemed that the best colored oil was obtained when using about 30 cc.,of a 10 degree Baume' solution of caustic Soda. The following data was obtained by using the weight of NaOH in 30 cc., of a 10 degree Baume' solution of NaOH or about 1.97 grams NaOH. The caustic was added to the oil in solutions on different strengths, namely, 14, 16 and 20 degrees Baume'. The nature of the precipitated soap stock was observed and also the

color of the oil obtained in each case.

SAMPLE #5.

Weight of oil used --------460 grams.

Strength of caustic used -- 14 degrees Baume'.

Volume of caustic used ---- 20.7 cc.

Conditions under which run was made;

The caustic solution was added to the oil when at a temperature of 25 degrees C. The two were agitated for about twenty minutes and then heated slowly to 45-47 degrees C., and held at this temperature for two hours.

SAMPLE #6.

Weight of oil used --------460 grams.

Strength of caustic used -- 16 degrees Baume'.

Volume of caustic used ---- 17.8 cc.

The conditions under which the run was made were the same as for Sample #5.

SAMPLE #7.

Weight of oil used --------460 grams.

Strength of caustic used --20 degrees Baume'.

Volume of caustic used ----13.7 cc.

The conditions under which this trial was made were the same as for Sample #5.

The precipitate in each of the above three trials was firm and settled rapidly. The indications were that the stronger solution of caustic gave a firmer and more coherent soap stock.

From the results of the trials which were made, it seems that when a low temperature is used in refining the oil, the soap stock produced was firm and settled rapidly, leaving a clear oil. Lower refining temperatures tend to produce and oil of better color. The color was also better when a greater amount of caustic, than the calculated amount was used. The color

appeared better when stronger solutions of caustic were used. When dilute solutions of caustic were used, an emulsion seemed to form whichwould probably be difficult to remove.

The effect of high temperatures was to saponify some of the oil, giving a soap stock which settled from the oil very slowly. The limiting temperature which gave a firm soap stock in the trials made, was from 40-47 degrees Centigrade. This temperature may vary with oils with different percentages of free fatty acid, but in the work done here only one oil was available and the results obtained and conclusions drawn are for that particular oil.

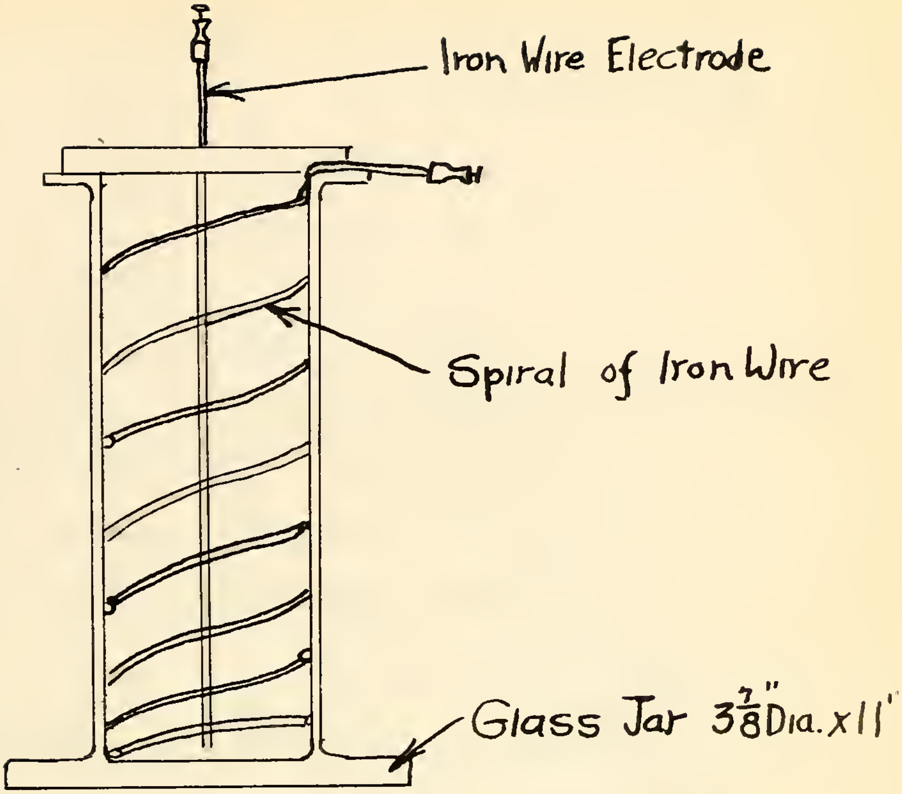

Fig II

TYPES OF CELLS
USED FOR ELECTROSTATIC PRECIPITATION

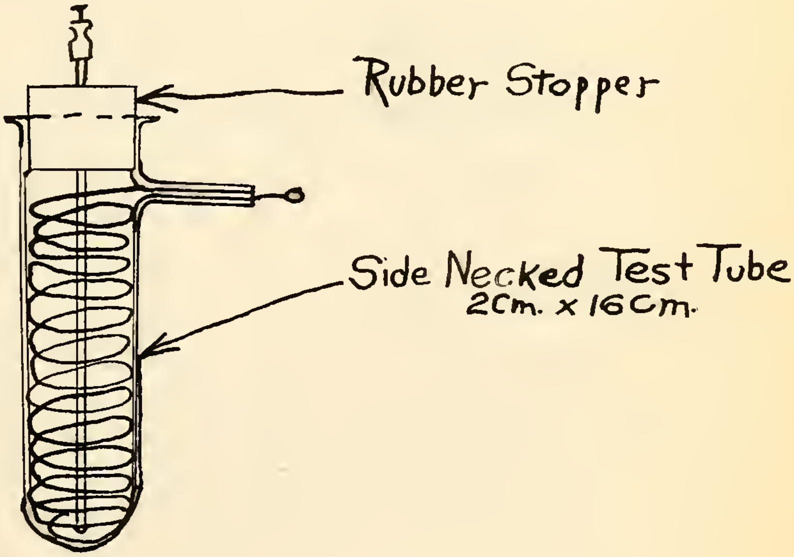

Fig III

## ELECTROSTATIC PRECIPITATION.

The first cell used, in attempting to solve the problem of precipitating the soap stock from the oil electrostatically, was made of a glass cylinder ten centimeters inside diameter and twenty-eight centimeters high. One electrode was a spiral of one-eighth inch iron wire, wound to fit just inside the glass cylinder. The other electrode was a straight piece of one-eighth inch iron wire, extending down through the center of the cylinder, at equal distance from the spiral electrode at all points. The two electrodes were connected across the source of current when making the experiments.

The apparatus used to give the difference of potential across the elctrodes consisted of a Rotary-Converter, which changed the available

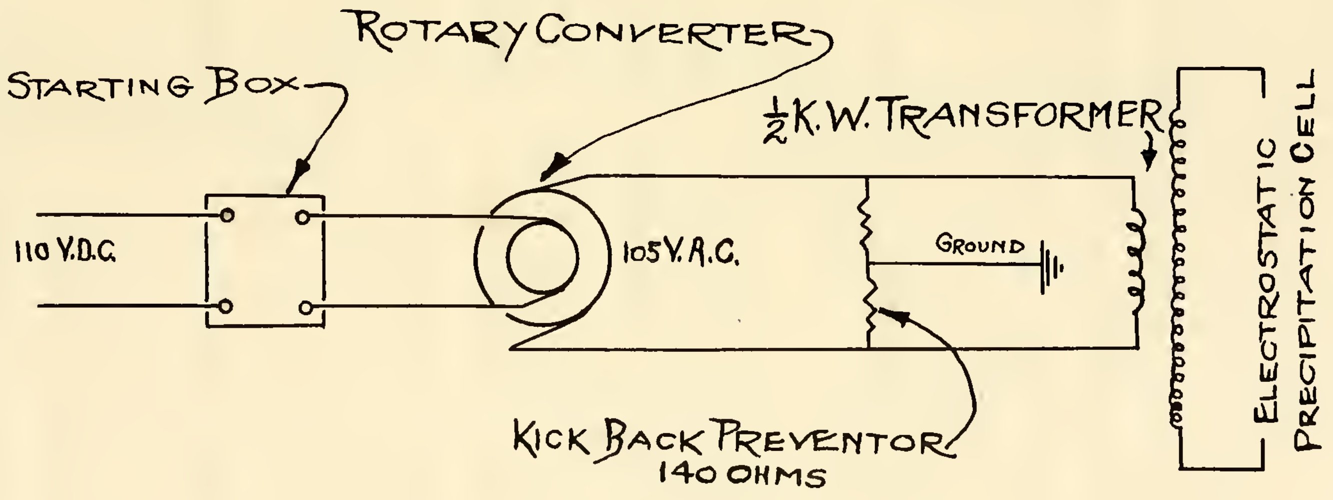
ROTARY CONVERTER
STARTING BOX
110 V.D.C.
105 V.A.C.
½ K.W. TRANSFORMER
GROUND
ELECTROSTATIC PRECIPITATION CELL
KICK BACK PREVENTOR
140 OHMS

FIGURE 4.

110 volt direct current to about 105 volts alternating current, a wireless transformer which changed the 105 volts from the converter to approximately 10,000 volts alternating current, a kick back preventer and a starting box for the Rotary-Converter. The wiring diagram is shown in Figure 4.

The two electrodes, of the cell, were connected across the terminals of the transformer to operate it. An attempt, to precipitate the soap stock from the oil, was made by filling the glass jar with freshly refined oil before any of the soap stock had settled and then passing the current through the oil. Observations showed that the current had no effect upon the soap stock in the oil and there was no movement of the soap stock particles. Different voltages were obtained by regulating the adjustable

shunt on the transformer but none of them had any effect upon the soap stock. The center electrode was moved close to the spiral electrode, just beyond sparking distance but no change was noticed in the oil. The straight wire electrode was then moved within sparking distance of the spiral and the only change noticed was the evolution of a gas from the oil.

The evolution of the gas was probably caused by a decomposition of the caustic solution used in the refining. It could be stopped by decreasing the voltage through the oil. Suitable apparatus to determine at what voltage this decomposition took place was not available so the exact voltage at which decomposition took place could not be determined.

An induction coil was now used in place of the transformer. The current for the coil was obtained from a battery of storage cells so arranged that they could be connected in any combination of series or parallel. The cell described above was used with the coil and from observations, when the electrodes were close together, it seemed that they were affected by the current passing through the cell. A difficulty seemed to arise from the fact that the cell used was too large for the voltage obtained from the induction coil. A new cell was made up of a smaller dimension so that all parts of the oil would be within the electrostatic field.

The new cell used was made of a side-necked test tube, two centimeters in diameter and sixteen centimeters long. One electrode was a spiral of light iron wire

fitted to the inside of the tube. The second electrode was a straight piece of iron wire passing down through the center of the tube cover and spiral wire. The purpose in using this form of tube was to overcome sparking between the electrodes along the part which was not covered with oil. With an ordinary tube, sparking occurred above the oil and this was overcome by passing one electrode through the side opening in the tube and keeping the oil level above this opening.

The freshly refined oil was poured into the tube to a level above the place where the spiral wire passed from the tube. The electrodes were connected across the secondary of the induction coil and the current was passed and the results were noted. Variations of voltage were obtained by arrang-

ing the cells in different combinations of series and parallel. The effects of each trial were noted and the results recorded.

An interesting fact was observed in the case of both cells. When the freshly refined oil was poured into the cells, before the soap stock had time to settle out, and the current was passed, the effect upon the transformer and induction coil was similiar to that of a short circuit. After the soap stock began to settle out and become less dense between the electrodes, a sparking occurred between the soap stock particles and the electrodes and the short circuit appeared to be broken. The conclusion drawn was that the soap stock must be a conductor of electricity and capable of bearing an electrostatic charge.

The small cell was used for all the remaining trials and the results obtained are recorded later.

## EFFECTS OF DIFFERENT VOLTAGES UPON THE SOAP STOCK IN THE OIL.

When single cells, in any number, were used, connected in parallel, the soap stock was not affected in any manner. Using the cells in this manner gave approximately two volts across the primary of the coil and an amperage dependant upon the number of cells used in parallel.

Two cells were connected in series and the discharge obtained across the secondary was estimated at about 8,000 volts from the length of the discharge between point electrodes through air. When the current was passed through the

cell, the soap particles seemed to concentrate around the electrodes and appeared more dense near the wire, the number decreasing as the distance from the wire became greater. The effect was similiar to that of iron filings when brought into a magnetic field. If the current were stopped for a few minutes, the soap stock particles started to settle to the bottom and when the current would again be passed they would concentrate around the electrodes. The soap stock which was suspended between the two electrodes, appeared to move back and forth for a small distance but would not move completly to one electrode or the other.

When three cells were used in series, the behavior of the soap stock was the same as when two cells were used. When four cells

were used in series, gas bubbles were formed, showing that decompsition of the water took place. The soap stock was affected some in each case but a complete precipitation was not accomplished. The voltage could not be increased any more because the discharge was too great for the cell used.

## CONCLUSION.

From the results of the experiments conducted, it seems likely that soap stock formed in an oil during the refining operation may be precipitated electrostatically in a prcperaly desighned cell and with current within certain limits of voltage. The results obtained under certain conditions seemed to indicate that a precipitation occurred and further study in to the problem might aid in solving the problem.

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